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Axial length effects on Lean NO_x Trap performance

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ABSTRACT

The effect of axial length on the NO_x reduction performance of two different commercial Lean NO_x Trap (LNT) monolithic catalysts was experimentally investigated in a bench flow reactor. The washcoat composition of one of the catalysts consisted of Pt and K on γ -Al₂O₃; whereas the other catalyst contained a complex mixture of Pt, Pd, Rh, Ba, Ce, Zr, Mg, Al and others. The NO_x removal characteristics of cylindrical monolith segments of constant diameter (2.22 cm) and axial lengths of 2.54, 5.08 and 7.62 cm were evaluated using a simulated lean engine exhaust containing water and carbon dioxide at a constant space velocity of $30,000 \, h^{-1}$. No significant effects of length were observed when the catalysts were fully reduced with hydrogen between NO_x capture phases. However when the catalysts were only partially regenerated NO_x reduction efficiency increased with monolith length. Intra-catalyst H_2 measurements at different axial locations indicated that at least some of the efficiency loss during partial regeneration occurred when back-mixed H_2 was directly oxidized and became unavailable for nitrate reduction.

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1. Introduction

In recent years, utilization of lean burn engines has increased because of their higher fuel efficiency and potential for lower emissions of hydrocarbons (HC) and CO. Unfortunately, NO_x removal for lean exhaust is problematic because the excess oxygen prevents use of the three-way catalyst technology that has become standard for controlling NO_x from stoichiometric engines. Several technologies have been proposed for lean NO_x abatement such as Ammonia-Based Selective Catalytic Reduction (NH₃-SCR), Hydrocarbon-Based Selective Catalytic Reduction (HC-SCR), Lean NO_x Catalysts (LNC) and Lean NO_x Traps (LNTs).

Current Lean NO_x Trap technology appears to have higher NO_x conversion efficiency and wider temperature limits compared to HC-SCR and LNC, and it does not require on-board storage of reductant chemicals as does NH_3 -SCR. In Lean NO_x Trap technology, NO_x reduction is achieved by first converting exhaust NO_x into solid nitrates under normal lean exhaust conditions and then reducing the nitrates during periodic rich exhaust excursions of short duration. A typical LNT catalyst consists of honeycomblike ceramic monolith coated with three primary components: precious metals such as Pt, Pd and Rh, alkali or alkaline-earth metals such as Ba and K dispersed on a high surface area support material such as γ -Al₂O₃. The precious metal components are important for oxidizing the NO component of NO_x to NO_2 , which

then reacts with the alkali/alkaline earths to form nitrates. During periodic rich excursions, NO_x is released from the storage sites and (ideally) reduced to N_2 over precious metals [1–6].

Emissions control engineers typically assume that catalytic reactor performance is determined by the space time (which is often expressed in its reciprocal form as gas hourly space velocity or GHSV) as long as all other conditions remain constant. This implies that as exhaust flow increases, it should be possible to maintain equivalent performance by proportionately increasing reactor length (or vice versa). If this assumption is correct for LNTs, one would expect that it should be possible to use this criterion in scaling LNT reactor length between different systems (or between measurements at different labs or a lab and vehicle). The validity of this assumption seems reasonable since monolithic LNTs are designed to operate in the laminar flow regime and turbulence is not a factor. However, significant discrepancies in LNT's performance are often obtained even though the evaluations were performed under the same conditions, i.e., same catalyst formulation, temperature, space velocity and simulated exhaust gas mixture composition [7]. As different sample sizes are often used in LNT evaluation due to different size of the reactors, length is suspected as potential cause of these disparities [7].

While much research has been focused on understanding NO_x storage and reduction chemistry [3,8–19], little has been done to assess the effect of length on the performance of Lean NO_x Traps. Thus the work described here was initiated to determine experimentally if and how monolith length might affect LNT performance independent of gas space velocity. The experimental approach involved detailed measurements of NO_x and hydrogen

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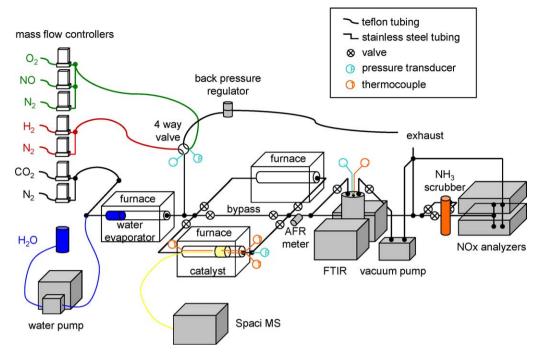


Fig. 1. Schematic of the bench flow reactor.

conversion for monoliths of different length in a bench flow reactor at a fixed gas hourly space velocity.

2. Experimental

A bench flow reactor illustrated schematically in Fig. 1 was used to measure the performance of different segments of the two LNT catalysts. Individual components of the simulated exhaust gases were introduced to the reactor via mass flow controllers, which were in turn connected to three separate gas manifolds. Simulated lean exhaust conditions were created by mixing NO, O₂, N₂, H₂O, and CO2. Simulated reducing conditions were created by mixing H₂, N₂, H₂O, and CO₂. For lean/rich cycling a solenoid-actuated four-way valve with fast switching times (0.1-0.2 s) was used to introduce the lean or rich gas flow into the reactor. Water was added to the inlet gases by means of a water evaporator and heated line. A peristaltic pump delivered liquid water to the evaporator, where the water was wicked onto quartz wool, evaporated, and then swept away by the inert portion of the exhaust gases. Directly downstream of the water evaporator, the wet inert gases were combined with lean or rich components and introduced into the reactor.

Individual segments of 2.22-cm-diameter catalyst monolith were wrapped in Zetex ceramic fiber strands to prevent gas bypass and then inserted in a 2.54 cm OD quartz-tube reactor, which in turn was placed inside a tubular furnace. The catalyst segment was typically positioned 25.0 cm from the quartz-tube inlet. The section upstream of the catalyst sample was filled with glass beads to promote mixing and uniform heating of the incoming gases. Three type-K thermocouples were used to measure the inlet and exit temperature as well as the temperature at the middle of the LNT catalyst sample, and a pressure transducer measured the reactor exit pressure.

Intra-channel hydrogen measurements were made using a Spatially Resolved Capillary Inlet Mass Spectrometer or SpaciMS [20]. The SpaciMS employs capillary probes of 185 μ m outer diameter for sampling the intra-channel gas phase for analysis. The sampling end of the capillary probe was inserted through the front

end of the reactor and positioned at different axial locations along the catalyst centerline. Following the usual SpaciMS configuration, the other end of the capillary was connected to a multiple-port valve leading to spectrometer's ionization chamber, and the sampling gas mixture was analyzed by a magnetic sector mass spectrometer.

Both inlet and exit gases from the monolith were analyzed with a MIDAC Fourier Transform Infrared Spectrometer (FTIR) and two California Analytical Instruments chemiluminescent NO_x analyzers. The FTIR equipped with a 375 mL and 3-m pathlength gas absorption cell measured the concentrations of NO₂, N₂O, NH₃, CO, CO₂ and H₂O. Upstream of the two California Analytical Instruments NO_x analyzers, an ammonia scrubber prevented NH₃ from interfering with NO_x measurements.

The two commercial LNT monoliths used in this study were both constructed from cordierite but had different washcoat formulations and cell densities. One of the monoliths with a cell density of 31 cells/cm² (referred herein as Catalyst A) is sold for use in gas turbine exhaust emissions control for electric power generation. The washcoat on this catalyst consists primarily of Pt and K supported on γ -Al₂O₃. The other catalyst, Catalyst B with a cell density of 97 cells/cm², is sold commercially for gasoline direct injection (GDI) engine applications. This catalyst has been provided by Umicore to the Crosscut Lean Exhaust Emissions Reduction (CLEERS) activity supported by the U.S. Department of Energy [21], and it is being widely used as a reference catalyst for LNT studies by CLEERS community [24-26]. The washcoat on Catalyst B contains platinum, palladium, rhodium, barium, cerium, zirconium, magnesium and aluminum as major constituents. Additional details of Catalyst A [22,23] and Catalyst B [24–26] can be found in previous publications.

For the experiments, three different lengths of both LNT monoliths (2.54, 5.08 and 7.62 cm long) were cored as 2.22-cm-diameter cylinders from full-size monoliths. Each of these segments was then subjected to lean-rich cycling at the conditions summarized in Tables 1 and 2. For all these experiments, the GHSV was held fixed at $30,000 \, h^{-1}$ by adjusting the gas flow for each different length segment of monolith. In the present study, the

Table 1Lean/rich cycling operating conditions for catalyst A.

Mode	Time	Gas composition
Lean Rich	56 s 4 s	300 ppm NO, 10% O ₂ , 5% H ₂ O, 5% CO ₂ , balance N ₂ 1 (or 2)% H ₂ , 5% H ₂ O, 5% CO ₂ , balance N ₂

 Table 2

 Lean/rich cycling operating conditions for catalyst B.

Mode	Time	Gas composition
Lean Rich	60 s 5 s	300 ppm NO, 10% O ₂ , 5% H ₂ O, 5% CO ₂ , balance N ₂ 1.4 (or 3.4)% H ₂ , 5% H ₂ O, 5% CO ₂ , balance N ₂

GHSV is defined as the ratio of volumetric flow rate at standard temperature and pressure to the volume occupied by the catalyst's outside dimensions. Each monolith segment was also assessed at three different temperatures.

Note that the only reductant used was H_2 , and two different concentrations of H_2 were added during the rich phases: a lower level to produce partial regeneration of the stored NO_x and a higher

level for full regeneration. The lower H₂ concentration was below the theoretical amount needed to completely reduce the integrated incoming NO_x during the trapping part of the cycle (lean phase). On the other hand, the higher H₂ concentration was considerably greater than the theoretical regeneration requirement. Such a high level was required because of the presence of stored oxygen in addition to stored NO_x (especially in Catalyst B). To determine the oxygen contribution it was necessary to make additional experimental measurements of oxygen storage capacity (OSC) in the absence of NO_x. This stored oxygen component contributes to the consumption of H₂ and thus must be combined with that needed to reduce the NO_x in order to accurately account for the amount of H₂ needed to reduce the catalyst. Operating conditions for these OSC experiments were similar to the conditions given in Tables 1 and 2, but without the presence of NO in the lean phase.

Another possible mechanism for H_2 consumption is the catalytic oxidation of H_2 at the interface between the lean and rich gas phases; the extent of which depends on the degree of mixing at the lean/rich interface. To account for this possibility, OSC experiments with a N_2 purge (5% H_2O , 5% CO_2 and balance N_2) of 10 s between lean and rich pulse (referred to as back-mixing

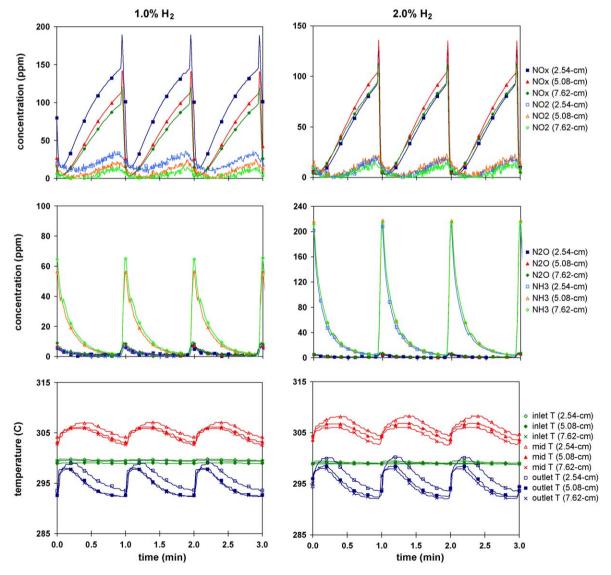


Fig. 2. Exit gas concentrations and temperature profiles for 2.54, 5.08 and 7.62-cm-long catalyst A samples in lean/rich cycling experiments with 1.0 and 2.0% H₂ in rich phase at 300 °C.

Table 3 NO $_x$ conversion efficiencies of Catalyst A in lean/rich cycling experiments with 1.0 and 2.0% H $_2$ in rich phase at 300 °C.

Concentration length	1.0% H ₂	2.0% H ₂
2.54 cm	65.7%	82.1%
5.08 cm	77.1%	79.5%
7.62 cm	80.5%	81.7%

experiments) were performed. N_2 purge creates an inert environment between the lean and rich front, which should eliminate H_2 consumption due to lean/rich front back-mixing.

Before any of the above experiments were conducted, Catalysts A and B were "degreened" to achieve stable and reproducible levels of their activity. Catalyst A was degreened by performing short storage-reduction cycles for 8 h at 550 °C; this degreening temperature is 150 °C higher than the highest operating temperature used in this study. In contrast Catalyst B was hydrothermally degreened in a furnace at 700 °C under a flow of 10% H₂O in air for 16 h as recommended by the manufacturer.

Prior to the evaluation, each catalyst segment was heated to $500\,^{\circ}\text{C}$ and subjected to a rich pulse consisting of $1\%\,H_2$, $5\%\,H_2$ O, $5\%\,CO_2$ and a balance of N_2 for 15 min in order to obtain an initially clean catalyst surface. The catalyst was then cooled to the operating temperature for the planned experiments.

3. Results

3.1. Catalyst A

Catalyst A was evaluated at three different temperatures (200, 300 and 400 °C), and two reductant concentrations (1.0 and 2.0%), and results were compared between 2.54, 5.08 and 7.62-cm-long samples. Fig. 2 shows the catalyst outlet gas concentrations, temperature profiles, whereas Table 3 lists cycle-averaged NO_x conversion efficiencies at 300 °C. A total of 120 cycles were performed but only 3 cycles recorded toward the end of the 120-cycle run are shown in the figure. Similar profiles were obtained at 200 and 400 °C. The cycle-averaged NO_x conversion efficiency was highest at 300 °C at a given reductant concentration. Also, the difference in performance was largest at this temperature; therefore, all of the data presented here is for 300 °C case.

As seen in Fig. 2, no significant difference was observed between 2.54, 5.08 and 7.62-cm-long samples with 2% H $_2$ in regeneration phase (full regeneration). However, a difference of 15% was observed between 2.54 and 7.62-cm-long samples using 1% H $_2$ (partial regeneration). On the other hand a difference of only 3.4% was observed between 5.08 and 7.62-cm-long samples with 1% H $_2$, which is within the experimental uncertainty; and thus it is inconclusive to attribute this difference to variation in length in these two samples. In addition, the level of NH $_3$ produced during the regeneration phase with 1% H $_2$ in 5.08 and 7.62-cm-long

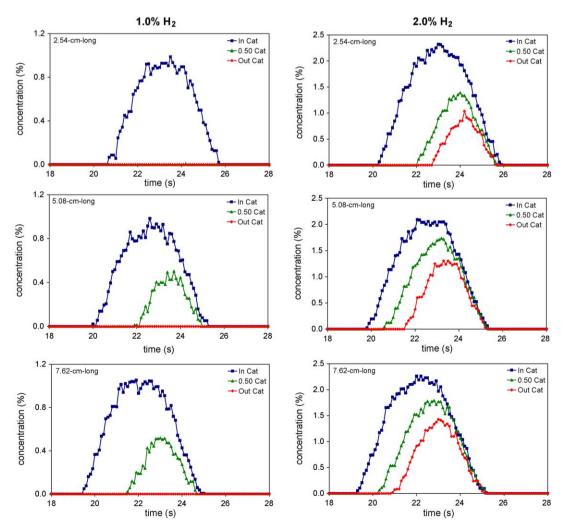


Fig. 3. H₂ consumption in 2.54, 5.08 and 7.62-cm-long catalyst A samples in lean/rich cycling experiments with 1.0 and 2.0% H₂ in rich phase at 300 °C.

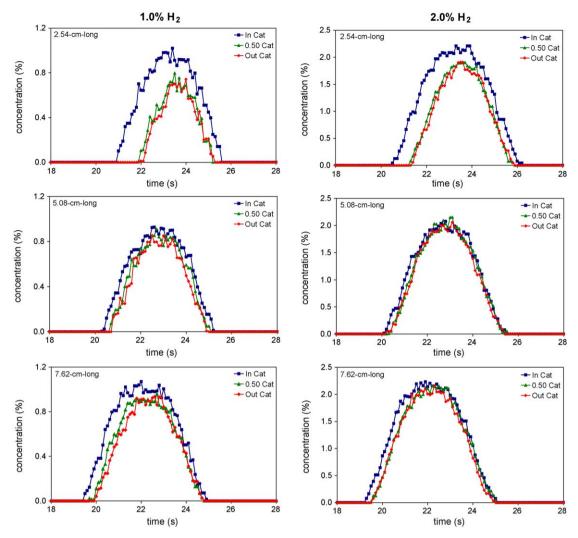


Fig. 4. H₂ consumption in 2.54, 5.08 and 7.62-cm-long catalyst A samples in oxygen storage capacity (without NO during lean phase) experiments with 1.0 and 2.0% H₂ in rich phase at 300 °C.

samples was also very similar but significantly larger than in 2.54-cm-long sample.

The intra-catalyst concentration of H_2 was measured at three different axial locations – catalyst inlet, mid-section and exit – with SpaciMS. Fig. 3 shows H_2 concentration profiles obtained at 300 °C, from which information on the consumption of H_2 along the length of samples of 2.54, 5.08 and 7.62-cm-long can be inferred. As seen in Fig. 3, with partial regeneration, i.e., 1% H_2 in the rich phase, H_2 was completely consumed in the front half of the 2.54-cm-long sample, whereas about 25% of H_2 remained in the 5.08 and 7.62-cm-long samples. With 2% H_2 in the rich phase, 80% was consumed in the entire length of 2.54-cm-long sample compared to 60% in 5.08 and 7.62-cm-long samples.

Results of OSC experiments are shown in Fig. 4. With 1% H₂ in the rich phase 50% of H₂ was consumed in the front half of the 2.54-cm-long sample and 18% in the 5.08 and 7.62-cm-long samples; furthermore, no significant H₂ consumption was observed in the rear half of the catalyst regardless of the length of the sample. Similar results were obtained in OSC experiments with 2% H₂.

Fig. 5 shows results of back-mixing experiments. With the introduction of a 10-s N_2 purge between the lean and rich phases, H_2 consumption in the front half of the 2.54-cm-long sample decreased by 35% in experiment with 1% H_2 in rich phase and by about 10% in both 5.08 and 7.62-cm-long samples. As a result of N_2

purge the H_2 consumption became very close regardless of the sample length. Similar results were obtained with $2\%\ H_2$.

3.2. Catalyst B

The evaluation of Catalyst B was carried out at 230, 325 and 500 °C using similar lean/rich cycling employed in evaluating Catalyst A. Data presented here is for 325 °C case, which was the temperature where highest cycle-averaged NO $_{x}$ conversion efficiency was obtained. General trends for the 230 and 500 °C cases are similar to the 325 °C case. Fig. 6 shows the catalyst outlet gas concentrations and temperature profiles, whereas Table 4 lists cycle-averaged NO $_{x}$ conversion efficiencies for lean/rich cycling experiments with 1.4% and 3.4% H $_{2}$ in the rich phase at 325 °C. A difference of 10% was observed between 2.54 and 5.08-cm-long samples with 1.4% H $_{2}$ in rich phase and 8% between 5.08 and 7.62-cm-long samples. Increasing H $_{2}$ concentration from 1.4 to 3.4% in rich phase resulted in nearly 100% NO $_{x}$ conversion for all samples.

Consumption of H_2 along the length of 2.54, 5.08 and 7.62-cm-long samples with 1.4 and 3.4% H_2 (results not shown in this manuscript) followed the previously observed trend in Catalyst A: more H_2 was consumed in shorter sample. For example, at 325 °C and 1.4% H_2 in the rich phase, H_2 was completely consumed in the front quarter of the 2.54-cm-long sample, whereas about 23% of H_2

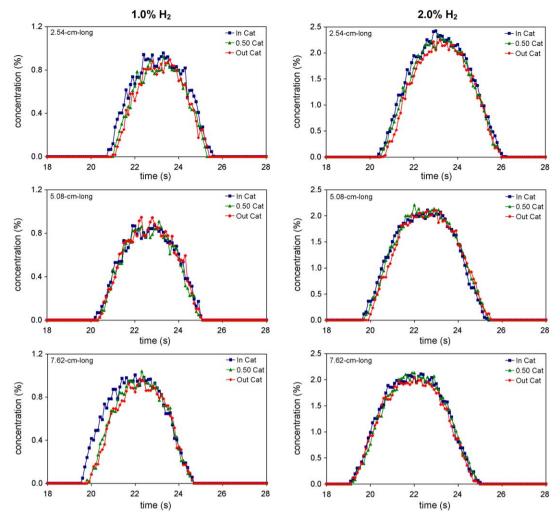


Fig. 5. H₂ consumption trends in 2.54, 5.08 and 7.62-cm-long catalyst A samples in back-mixing (OSC experiments with 10 s nitrogen purge between lean and rich pulses) experiments with 1.0 and 2.0% H₂ in rich phase at 300 °C.

remained in the 5.08 and 14% in 7.62-cm-long samples. Experiments without NO in the lean phase, similar to those performed on Catalyst A (OSC experiments) and OSC experiments with a N $_2$ purge (back-mixing experiments) were also carried out. These experiments led to H $_2$ consumption trends similar to those obtained on Catalyst A. However, quantitative data analyses were difficult for this catalyst due to its high OSC content. In fact, significant amount of the stored oxygen and possibly H $_2$ desorbed during the N $_2$ purge and made quantitative comparison challenging.

4. Discussion

Consumption of H_2 in lean–rich cycling experiments with two different concentrations of H_2 in the rich phase followed the same trend: more H_2 was consumed in 2.54-cm-long sample than in 5.08 and 7.62-cm samples. This higher consumption of H_2 in 2.54-cm-long sample could be interpreted, a priori, to indicate a higher NO_x conversion, but the opposite was observed. A lower NO_x conversion was obtained with 2.54-cm-long sample than with longer samples for the partial regeneration cases, and no difference was observed for the full regeneration cases as shown in Figs. 2 and 6. The lowest H_2 consumption was also obtained with 2.54-cm-long samples during OSC experiments. Considering equivalent amount of NO_x and oxygen stored per volume of the catalyst during lean phase for all different length samples, these opposite trends

seem to suggest that there were H_2 consumption mechanisms other than NO_x and OSC reduction.

The catalytic reaction between H_2 and O_2 at the interface between the lean and rich phases may have contributed significantly to H_2 consumption; the extent of which depended on the degree of mixing at the lean/rich interface. Higher degree of back-mixing should result in higher H_2 consumption and lesser amount of H_2 available for reducing stored NO_x , thereby lowering catalyst's NO_x conversion. Therefore higher consumption of H_2 in 2.54-cm-long sample than in 5.08 and 7.62-cm samples may be attributed to higher degree of back-mixing in a shorter sample; whereas lower H_2 consumption in longer samples apparently indicates that lesser degree of back-mixing was achieved when the flow rate was increased to the levels used in longer samples (at a fixed gas hourly space velocity and catalyst diameter, gas velocity or flow rate increases with increasing sample length).

Based on OSC experiments with a N_2 purge (Fig. 5), it is indeed clear that significant amount of H_2 was consumed by the catalytic reaction between H_2 and O_2 at the interface between the rich and lean pulses. Slower moving gases resulted in higher degree of mixing at the lean/rich interface (as in the case of 2.54-cm-long sample), which culminated in higher H_2 consumption and lesser amount of H_2 available in reducing NO_x . As the flow rate reached a certain level, back-mixing became negligible (as in the case of longer samples) and no difference in catalyst performance was

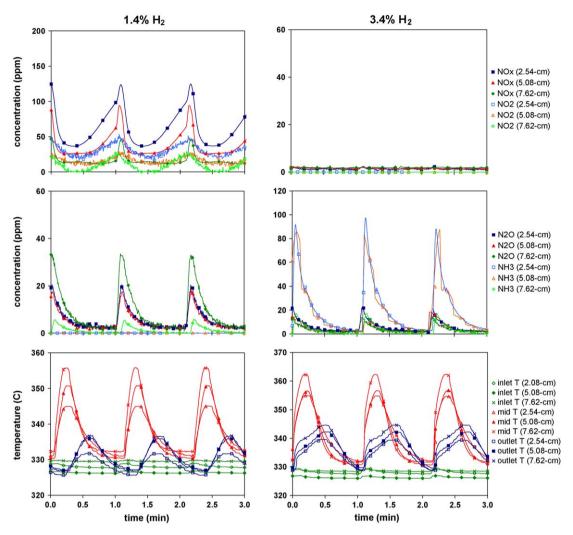


Fig. 6. Exit gas concentrations and temperature profiles for 2.54, 5.08 and 7.62-cm-long catalyst B samples in lean/rich cycling experiments with 1.4 and 3.4% H₂ in rich phase at 325 °C.

observed. For example, in the 2.54-cm-long Catalyst A sample as much as 35% of H_2 was consumed from the catalytic oxidation at the lean/rich interface – compared to only 10% in the longer sample – resulting in a reduction of 15% in NO_x conversion with 1% H_2 (Figs. 4 and 5, Table 3). Assuming that for every mole of NO entering the reactor 2.5 moles of H_2 are required to reduce the stored NO_x to N_2 , 25–35% less availability of H_2 would result in 10–14% decrease in NO_x conversion when regeneration is limited by amount of H_2 available. Therefore, the observed NO_x conversion differences for samples of different lengths with partial regeneration match well the corresponding differences in H_2 availability within experimental uncertainty. Lower formation of NH_3 in 2.54-cm-long sample than in longer samples further indicates lesser availability of H_2 in shorter sample, since NH_3 production decreases with decreasing H_2 concentration [26–29].

Table 4 NO_x conversion efficiencies of Catalyst B in lean/rich cycling experiments with 1.4 and 3.4% H₂ in rich phase at 325 °C.

Concentration length	1.4% H ₂	3.4% H ₂
2.54 cm	75.3%	99.5%
5.08 cm	85.1%	99.4%
7.62 cm	93.1%	99.2%

5. Conclusions

The effect of monolith length on the NO_x performance of two different Lean NO_x Traps was investigated in the present study. The evaluation was carried out on samples of three different lengths with lean/rich cycling at a fixed gas hourly space velocity. No significant difference in performance was observed during lean/rich cycling with full regeneration. On the other hand, large discrepancy was observed with partial regeneration: the longer the sample the better the performance.

 $\rm H_2$ consumption trends during evaluation indicated different degrees of lean and rich front back-mixing in samples of different lengths. Higher back-mixing resulted in a higher $\rm H_2$ loss via its oxidation by $\rm O_2$ and lesser $\rm H_2$ availability for reducing stored $\rm NO_x$, which in turn affected catalyst's performance when regeneration was limited by amount of $\rm H_2$ available.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.apcatb.2009.07.030.

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